LINDE DIVISION
UNION CARBIDE CORPORATION
Speedway Laboratories
P. O. Box 24184
Indianapolis, Indiana

First Quarterly Progress Report

FABRICATION AND PROPERTIES OF

TUNGSTEN SINGLE CRYSTALS

"Growth of Single Crystal Tungsten

To Be Used for Rolling Into

Single Crystal Sheet"

Work done and reported by: F. R. Charvat
                                                    G. W. Edwards

For Period March 1 to April 30, 1964

Prepared Under Contract NOw 64-0055-c

For the Materials Branch,
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SUMMARY:

This is the first quarterly report covering the work done for the Materials Branch, Bureau of Naval Weapons under Contract Number NOw 64-0055-c and covers the period from March 1 to May 31, 1964.

A new set of master seeds were produced and oriented so that the [011] and [001] crystallographic direction would coincide with the cylindrical axis of the crystal. These seeds were prepared similarly to those used in the previous program. The master seeds of [011] and [001] orientation used in the previous program will be used to complement the seed stock used for this program.

Two crystals of inferior quality were machined and sent to Aeronutronics Division, Philco Corporation to be used to review their rolling technique and for preliminary studies on rolling temperatures.

Ten tungsten crystals were grown by the arc-verneuil process from which three were selected on the basis of minimum crystal misorientation and deviation from cylindrical axis. Chemical analysis for interstitial and metallic impurities of these crystals has not yet been completed.

The three crystals selected were machined to form rectangular billets to allow uniform deformation and crystallographic rolling plane and rolling direction to be within the desired limits.

The continued effort for the second quarter will be to produce the necessary oriented single crystal billets designated by Aeronutronics to successfully complete this portion of the program.

I. INTRODUCTION

Experimental data obtained from the previous investigation show that it is feasible to deform single crystal tungsten by roll-anneal cycles and still retain its monocrystallinity. It was also shown that certain crystallographic orientations, specifically the (100)(011) and (110)[001] orientations, behave in superior fashion to others. Thus, by using properly oriented crystals, large single-crystal sheet could be obtained by employing appropriate consecutive deformation-anneal cycles.

As in the previous investigation, the portion of work under the contract to be performed by the Linde Division at Speedway, Indiana, consists of growing single crystals of tungsten or its alloys, evaluating the grown crystals as to their perfection and orientation, and fabricating them into billets suitable for rolling. Philco Division will then work the billets by rolling, etc., and perform the evaluation on the rolled product.

II. MASTER SEED PREPARATION

As was noted in earlier work, in order to grow high quality tungsten single crystal via the arc-verneuil method, it is imperative that seed stock be of highest obtainable quality. The two crystals used for seed stock, [011] and [001] orientations, were ground to smooth cylinders having one end normal to the crystal axis. This end was then electrolytically polished in a 10% sodium hydroxide using a high current density to remove distorted material caused by the machining and grinding. A Laue Back Reflection pattern was obtained and the necessary corrections were made to bring the cylinder axis and the desired growth direction in coincidence. Necessary machining techniques were then used to produce a sufficient length of the desired orientation. The seeds were then repolished and checked by Laue Pattern to ascertain final orientation.

III. CRYSTAL GROWTH AND BILLET PREPARATION

The seed crystals prepared above were used to grow ten oriented crystals of 7/16-inch diameter by 8-inches long, listed in Table 1. Three crystals were selected from this group to be processed into billets 3/8-inch wide by 1/4-inch thick by 6-inches long for rolling. The major reason for rejection of the remaining crystals was the presence of low angle grain boundaries. Billet preparation procedure described previously[1] was used to prepare the three crystals selected. Two as-grown crystals and two machined billets are shown in Figure 1.

These three billets will be shipped to Aeronutronics as well as a copy of the Laue and Schulz-Wei Pattern for each when final X-ray and chemical analyses are complete.

GW Edwards

G.W. Edwards:nm

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TABLE I.

| Crystal Number	Rolling Plane	Rolling Direction	Comments
1690-20		[011]	Rejected because of excessive low angle grain boundaries.
1699-21	-	[100]	"
1699-22	(011)	[100]	Accepted for rolling.
1699-23	-	[011]	Rejected because of excessive low angle grain boundaries.
1699-24	(011)	[100]	Accepted for rolling.
1699-25	-	[011]	Rejected because of excessive low angle grain boundaries.
1699-26	-	[011]	"
1699-27	-	[011]	"
1699-28	(100)	[011]	Accepted for rolling.
1699-29	-	[011]	Rejected because of excessive low angle grain boundaries.
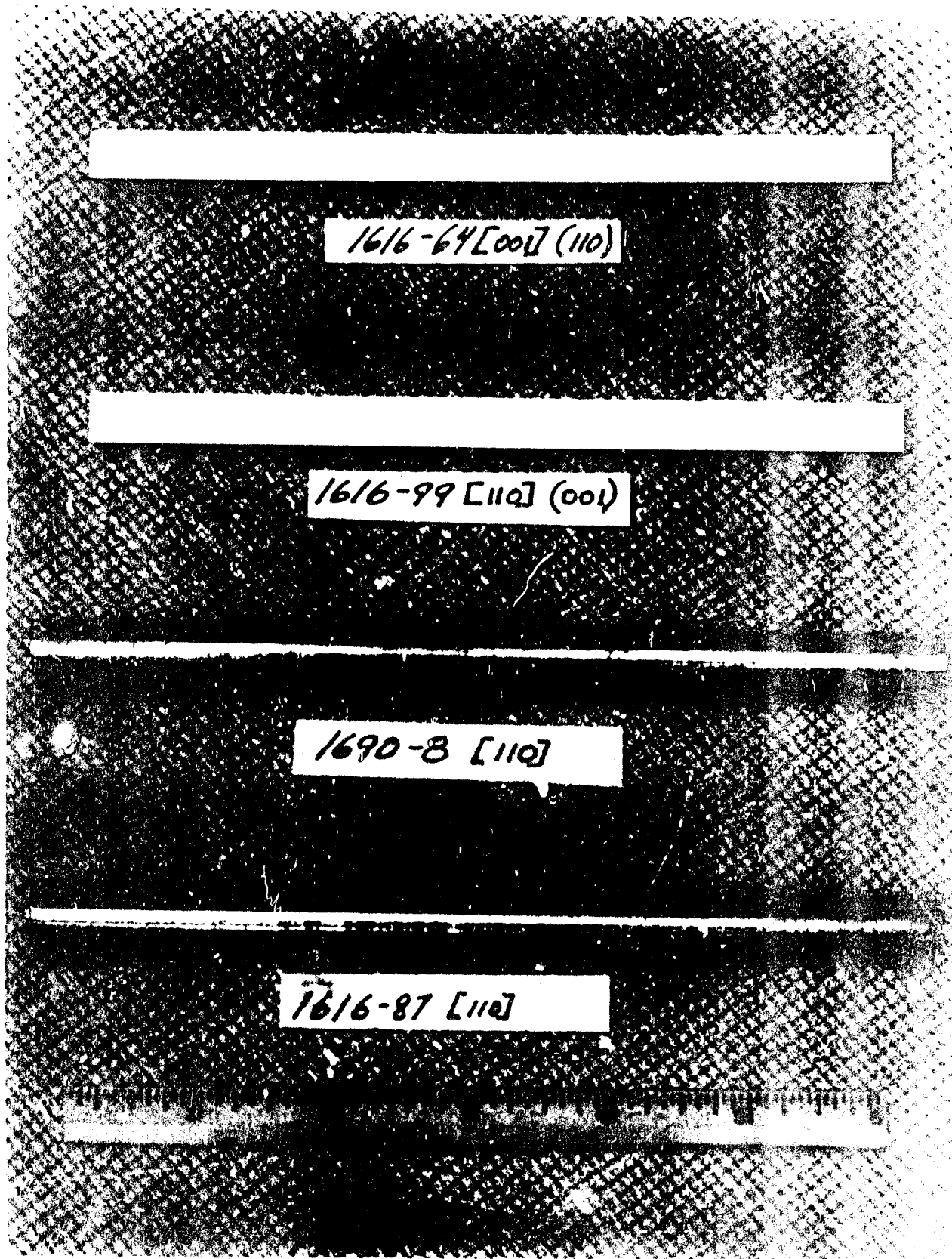
Figure 1.   Tungsten Crystals As-Grown and
             Tungsten Crystals After Machining

PHILCO
A Division of Ford Motor Company,
RESEARCH LABORATORIES

QUARTERLY TECHNICAL REPORT

SINGLE CRYSTAL TUNGSTEN SHEET

Reporting Period:	23 January - 30 April 1964
Prepared for:	Linde Company, A Division of Union Carbide Corporation Indianapolis, Indiana
Under Contract:	Subcontract No. 1 to Prime Contract NOw 64-0055-c
Prepared by:	L. Raymond

30 May 1964

ABSTRACT

(from page 1 first report, included in front)

Should: New paragraph

The program is designed to illustrate both the ability and the feasibility of fabricating a large single crystal into sheet. Subsequent roll-anneal cycles will be used which retain monocrystallinity and thus the inherently low ductile-to-brittle transition temperature of tungsten single crystals. The previous program has illustrated the possibility of such an approach. This program is concerned with rigorously defining the variables which influence recrystallization of a tungsten single crystal in hopes to control and best utilize the variables in order to allow more flexibility in processing. The substructural changes accompanying the processing will then be observed and the effect of these changes on the ductile-to-brittle transition temperature recorded.

CONTENTS

INTRODUCTION

Because the increased temperature requirements of many rockets exceed the capabilities of molybdenum, tungsten has taken over many of the molybdenum applications and is the primary metal in terms of strengths above $3500^{\circ}F$ ($1930^{\circ}C$). The two principal problems related to polycrystalline tungsten are its lack of oxidation resistance and its high ductile-to-brittle transition temperature which results in the material being extremely brittle at room temperature. Alloying--both dispersion and solid solution--is being investigated at many laboratories throughout the United States in an attempt to increase both the high temperature strength and low temperature ductility. The attempts to date have not been completely successful.

A single crystal of tungsten, on the other hand, is extremely ductile at room temperature with a transition temperature reported as low as $-107^{\circ}C$. Its room temperature yield strength encompasses that of polycrystalline tungsten, ranging from 49 to 140 ksi depending on orientation. Hence, single crystals of tungsten inherently possess required room temperature ductility and competitive strength levels. The single crystal is also equally susceptible to strengthening by dispersion or solid solution.

With these facts in mind, it was decided to take advantage of the excellent inherent properties of tungsten single crystals and investigate both the ability and the feasibility of fabricating single crystal sheet from the large as-grown single crystals. The means by which single crystal sheet can be fabricated depends on the ability to remove the effects of the initial amount of work prior to the onset of recrystallization so that subsequent passes may be taken to increase the planar dimensions of the sheet at the expense of reducing the

thickness of the sheet. In order to do this a high recrystallization temperature is necessary so that high recovery temperatures can be used which would make the recovery time of a magnitude which is a realistic or practical value for annealing. If this process is shown to be reasonable and to provide the single crystal sheet with the inherently low ductile-to-brittle transition temperatures reported for single crystals,. then alloy strengthening of the single crystals would be considered with due regard to the accompanying changes in the recrystallization kinetics of the crystal.

PREVIOUS PROGRAM REVIEW

Under the previous subcontract, No. 1 under NOw-61-0671-c, an etch pit technique was utilized to observe changes in substructure for several crystal orientations as a function of deformation and subsequent annealing treatments. Complete recovery of the substructure, i.e., the return of a low or original dislocation density, was obtained for the (100) $[01\bar{1}]$ crystal after 3-to-5 per cent reduction in thickness by annealing at $2500^{\circ}C$. The (110) $[00\bar{1}]$ crystal, although not investigated as thoroughly, suggested a similar behavior. The remaining orientations, the (100) $[001]$ and the (111) $[\bar{1}1\bar{2}]$, displayed recrystallization tendencies even for small amounts of deformation.

The difference in behavior of the two sets of crystals was resolved by the tendencies of the favorable orientations for simpler modes of slip than the unfavorable orientations which were immediately subjected to multiple-slip because of crystal orientation. Under multiple slip, tangles of dislocations were produced early in deformation which on annealing, presumably coalesced to form polygonized subgrain boundaries which grew into high angle boundaries. Simple slip produces more orderly dislocation networks which begin to form tangles at larger amounts of strain than under the conditions of multiple slip. Above these critical amounts of strain, recrystallization occurs as described above, but below the critical amount of strain, complete recovery of the substructural density can occur without recrystallization.

It is below this critical deformation level that a (100) $[01\bar{1}]$ crystal was subjected to three consecutive rolling and subsequent annealing cycles. The original dislocation density was maintained after a deformation of about 10 per

cent and a final 30-minute 2500°C anneal whereas a similar amount of deformation on one pass would recrystallize at 1650°C. Concomitant with these observations was the complete absence of the as-grown subgrain boundaries. It appeared that the strain necessary to remove the subgrain boundaries increased as the misorientation angle across the boundary increased.

The results suggest that large amounts of deformation can be obtained in single crystals by appropriate consecutive roll-anneal cycles. The major limitation appears to be the small amounts of deformation necessary to stay below the critical value which produces recrystallized grains during annealing.

PRESENT PROGRAM OBJECTIVES

The objectives of the present program are detailed as follows:

a. Since a 3-to-5 per cent reduction in thickness has been shown to produce a substructive which can be completely recovered without the onset of recrystallization, it is hoped that a more practical figure for actual rolling operations of 7-10 per cent reduction in thickness can be obtained by taking advantage of prior recovery annealing treatments.

b. A detailed study of the influence of prior recovery treatments on the changes in substructure for the two most favorable orientations, the (100) [011] and the (110) [001], will be conducted.

c. The specimens described in (b) will be deformed at temperature of 1000°C prior to annealing. Since lower deformation temperatures (400°C for example) tend to raise the recrystallization temperature, the best condition in (b) will be reproduced for a lower deformation temperature, say 550°C. The reason for the caution employed in choosing a lower deformation temperature is that the increased recrystallization temperature results from the appearance of deformation twins which would be detrimental to the objective of the program.

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d. Once the optimum prior recovery annealing treatment is defined for the most favorable orientation, the treatment will be used on an orientation which has a low recrystallization temperature {(001) [100], for example} to see to what degree the same beneficial effects are obtained.

e. Under the above optimum defined conditions, consecutive roll and anneal cycles will be used to try and obtain a total deformation of about 80 per cent reduction in thickness.

f.. Under the above optimum conditions, deformation under the more complex condition of 90° cross-rolling will be used to take advantage of the orthogonal symmetry of the system in order to obtain a sheet both longer and wider than the original dimensions. Again the consecutive roll and anneal method will be used to obtain a total deformation of about 80 per cent reduction in thickness. In this manner, if successful, an original 1 x 2 inch specimen would result in an approximate .034 x 2½ x 5 inch specimen from a ¼ inch thick sample.

g. Since alloying elements, metallic more so than nonmetallic, tend to raise the recrystallization temperature, a W - 0.6Nb alloy single crystal of the most favorable orientation for the high purity crystal, will be subject to the same conditions as (d) for comparison.

h. Since substructural changes influence the D-B transition temperature and possibly the mechanical properties, advantage will be taken of X-ray diffraction, tensile tests and possibly bend tests in order to evaluate the influence of processing on the substructure and resulting mechanical properties.

i. Such factors as (1) X-rays to determine if simple glide rather than multiple glide is operative, (2) etch-pits to determine the dislocation density gradient through the thickness and (3) hardness versus recovery time measurement, will be made wherever necessary.

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QUARTERLY WORK STATEMENT

The first part of this program was to rigorously define the variables of the problem based on past and present knowledge. Current and past literature was used as a basis for selecting the process variables which were directed toward the critical aspects of the problem. In this way, it is hoped that the more practical aspects of the program, such as the feasibility study of sheet rolling, can be evaluated by fully utilizing applicable information presented by other laboratories.

QUARTERLY RESULTS

To devise a means by which a single crystal can be deformed and retain its monocrystallinity after annealing necessitates a complete understanding of the recrystallization kinetics and the variables which influence them. As classically observed, the recrystallization temperature (R_xT) is strongly dependent on the amount of deformation, decreasing as the amount of deformation increases. For a single crystal, this behavior is also a function of crystal orientation. Once the most favorable orientation is obtained i.e., the one which possess the highest R_xT for a given amount of deformation, other factors may also be considered in raising the R_xT. These are (a) prior recovery annealing (b) impurities (c) deformation temperature (d) applied recovery stress and (e) specimen thickness. The over-all behavior is schematically illustrated in Figure 1.

1. PRIOR RECOVERY ANNEAL
2. METALLIC IMPURITIES
3. LOWER DEFORMATION TEMPERATURE
4. EXTERNAL RECOVERY STRESS
5. REDUCED SPECIMEN THICKNESS
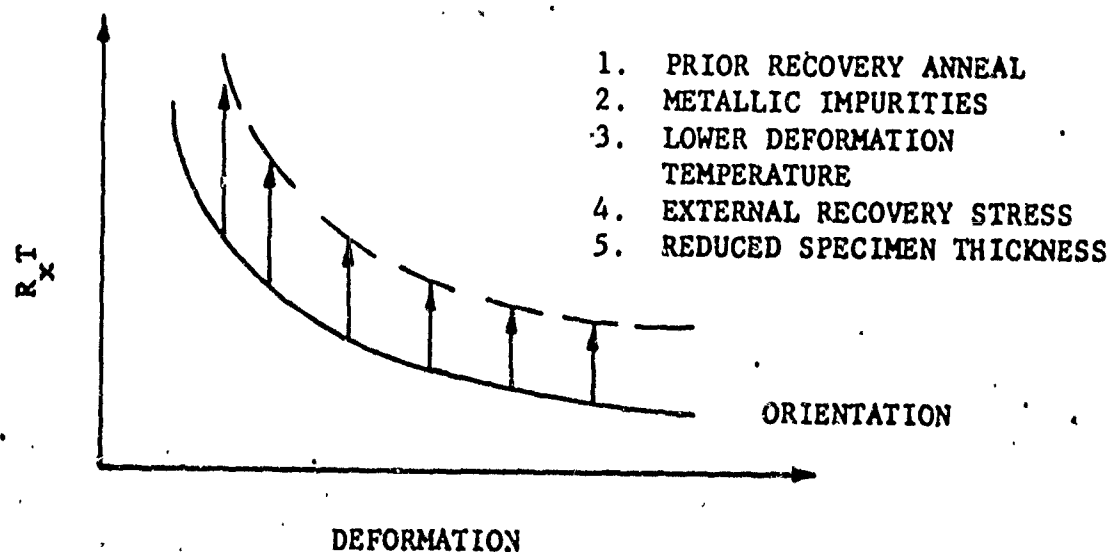
ORIENTATION

DEFORMATION

FIGURE 1. FACTORS WHICH RAISE THE RECRYSTALLIZATION TEMPERATURE

Selection of Process Variables

The basis for the selection of the process variables is the need for a high recrystallization temperature because the program emphasizes the recovery of the substructure without recrystallization. Therefore, in order to make the results of the program practical, the necessary recovery treatments should be based on a combination of high temperatures and short times.

a. Amount of deformation

Smithells[1] illustrates recovery of the mechanical properties without recrystallization in drawn tungsten single crystal wires. Provided that the reduction in areas has not been carried too far, the hardness may be removed without recrystallization taking place. This critical reduction varied from 50 to 80 per cent depending on the original diameter of the wire. The statement is made that for large single crystals, the smallest amount of deformation exceeds the critical value and recrystallization takes place on annealing.

There seems to be no a priori reason why the size of a single crystal should influence recrystallization. The results of the first program[2] show that 3 to 5 per cent deformation may be introduced into a 0.250 inch thick crystal and the work completely removed after a 2500°C, $\frac{1}{2}$ hour anneal. Subsequent roll-anneals may be used to increase the total amount of deformation without the onset of recrystallization. Since a 3-to-5 per cent reduction in thickness has practical limitations for actual rolling operations, an attempt will be to use 7-to-10 per cent reduction in thickness by taking advantage of prior recovery annealing treatments. This effect is also dependent on crystal orientation.

b. Orientation

The most favorable crystal orientation is the (100) [011] and possibly the (110) [001] .[2] This preference of orientation appears to be related to the type of slip operative for a given mode of deformation and crystal orientation simply because the operative slip systems are a function of the crystal orientation relative to the principal stresses. The differences are usually more detectable for smaller amounts of deformation. Larger amounts of deformation are normally accompanied by crystal rotation which removes any initial selection of operative slip systems. In general, simple slip is most conducive to recovery of the hardness without recrystallization. This has been shown[3]

in a silicon single crystal where the ease of polyonization depended on the orientation of the bending crystal. Vogel[3] found that if only one slip system is operative in silicon, a homogeneous aggregation of dislocations is produced which readily lends itself to polygonization but, if intersecting sets of dislocations are produced, polygonization is inhibited. Hence, initial orientation appears to influence the type of dislocation interactions which occur and, as a result, the ease with which they can recover without recrystallization on annealing. Evidence that the <110> tensile direction in tungsten single crystals results in plastic flow by simple slip is presented by Rose, Ferriss and Wulff[4] wherein the stress-strain curve for that direction is shown to approach an ideal elastic-plastic behavior with the strain hardening rate approximately zero.

In a single crystal of aluminum, Lutts and Beck[5] obtained complete recovery of the hardness prior to recrystallization. The crystal was oriented to provide the anticipated (110) [112] rolling texture which is also a predominant fcc recrystallization texture. In a bcc single crystal of Si-Fe[6] a (100) [011] oriented crystal had a stable texture during deformation. For the times and temperatures employed, the crystal did not recrystalize but polygonized. In both cases cited, [5,6] a heavy substructural density still remained after the annealing treatments employed. For the aluminum, after the hardness had been removed, recrystallization finally took place.

In general it appears that if the initial crystal orientation is chosen such that the deformation texture is identical to the recrystallization texture for that particular material, complete recovery of the hardness can be obtained prior to recrystallization. This rolling direction in tungsten single crystals appears to be the (100)[011] where the [011] is also the tensile direction of simple slip. It is worthy to note that recovery under these conditions occurs by subgrain boundary migration and dislocation density rearrangement but, a dense substructure is still maintained after complete recovery and prior to recrystallization .

The critical rolling texture must be in some way associated with a slip system which approaches easy glide and for tungsten single crystals. Assuming rolling to be a combination of compression and tension stresses, this condition should be obtained for some specific plane and the [110] rolling

direction. These observations are interesting and of similar nature to those under study but this application in the present investigation must be cautioned for not only is recovery of the hardness necessary but also complete recovery of the substructure in order to completely avoid recrystallization. For this reason the program must be limited to small amounts of deformation per pass and annealing treatments which remove both the hardness and the substructure. Larger amounts of deformation can only be used by taking advantage of prior recovery annealing treatments.

c. Prior Recovery Anneal

A limited amount of work[7] has been conducted on the influence of prior recovery on the recrystallization temperature of tungsten. A $100^{\circ}C$ increases in R_xT was reported[7] for a prior 10 minute anneal at $500^{\circ}C$. The same amount of prior recovery time at higher temperatures did not appear to raise R_xT. Other work[8] on single crystals of silver has shown prior recovery anneals to raise the R_xT. Specimen deformed up to 30 per cent by simple glide were found to be very sensitive to a prior recovery anneal. Above 30 per cent deformation long time prior annealing processes were necessary. These long time processes were also effective in specimens deformed 23 per cent in multiple glide. As an example of the long time prior annealing process the times and temperature employed were in the following sequence:

$$400^{\circ}C \text{ to } 470^{\circ}C \text{ in 1 hour}$$
$$475^{\circ}C \quad - \text{ 10 hours}$$
$$500^{\circ}C \quad - \text{ 10 hours}$$
$$600^{\circ}C \text{ - 60 hours}$$

Although the times involved are extensive, the interesting issue here is that under most complex slip systems up to 23% deformation where the R_xT is $500^{\circ}C$ prior recovery anneals resulted in no apparent recrystallization up to $850^{\circ}C$. The concluding comment is that for smaller amounts of deformation under conditions approaching simple glide, prior recovery anneal treatment should significantly influence the recrystallization kinetics of tungsten single crystals and should provide a promising approach for attaining complete recovery without recrystallization.

d. Impurities

Non-metallic impurities do not influence the recrystallization temperature (R_xT) as significantly as do metallic impurities.[9] What little effect,

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exists for the non-metallic is such that carbon raises the R_xT and oxygen and nitrogen decrease it. Metallic impurities have a much stronger effect.[9] The presence of about 25 ppm aluminum raises the R_xT by 300°C.[10] For these reasons, a metallic alloyed tungsten single crystals with only trace interstitial would be most suitable for the present investigation inasmuch as the interstitial impurities are very deleterious to the ductility. An alloy which has interesting high temperature properties and a high recrystallization temperature has been reported.[11] This alloy is W-0.6 Nb.

e. Deformation Temperature

Because of the accelerated effects of recovery with increasing temperatures, it would be generally expected that increasing the deformation temperature would increase the recrystallization temperature if this factor had any effect at all. Contrary to this opinion, a recent report[12] has shown lower deformation temperatures to raise the R_xT (½ hour in vacuum). Fifty per cent deformation in compression at 400°C recrystallized at 1900°C. The same amount of deformation at 1100°C recrystallized at 1200°C. This increase in R_xT was attributed to the appearance of twins at the lower deformation temperatures. Since twin interfaces can provide relatively high energy boundaries which behave in crack propagation similar to grain boundaries in polycrystalline materials,[13] the appearance of twins would be undesirable in attempting to retain the monocrystallinity of a single crystal after deformation. The appearance of twins would effectively be a grain size refinement. Therefore, for the purpose of this program, a higher deformation temperature should be maintained in order to avoid the undesirable complexities of twinning. Time permitting, a lower deformation temperature, say 550°C, will be used to evaluate this variable for the best available condition.

f. Stress Recovery

Although stress apparently plays a significant role in annealing, the effect must be considered lightly because of the practical limitations of stress-anneals. For the sake of completeness, the matter will be discussed briefly, because the comments should have application in evaluating high temperature properties. The reason for this interesting effect is suggested by the fact that recovery usually involves polygonization and subboundary migration[14] which is subjected to the conditions of migration illustrated by the Parker-Washburn

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experiment [15] where the velocity of a migrating tilt boundary is a function of the applied stress. The migrating boundaries, in turn, leave a "recrystallized structure" in their wake as illustrated by Rutter and Aust.[16] These factors suggest that an external stress should in some way effect the migration of sub-boundaries of the tilt type during recovery and its subsequent effect on recrystallization. Wood and Sutter[17] have illustrated this behavior in aluminum where recrystallization was inhibited with the application of a small external stress during annealing. Since both aluminum and tungsten have high stacking fault energies and both appear to have identical behaviors during recovery as identified by the electron microscope,[18] there is no reason not to believe that recrystallization in single crystals of tungsten can be inhibited by the application of a small external stress during annealing.

Some existing observations to substantiate the argument are the observations of Harmon and Forgeng[19] wherein migrating subboundaries have been observed in tungsten on annealing. Also, Wolff[13] has shown that these boundaries are usually tilt type boundaries. For a molybdenum alloy, the R_xT has been shown[20] to be greatly influenced by the presence of a tensile stress. Also, the recrystallization texture under stress was found to be different than in the stress-free anneal.

g. Specimen Thickness

As discussed in (a), the critical reduction increases as the specimen thickness decreases. Therefore, any optimum annealing treatments outlined for the initial pass, should be more than adequate as the thickness of the specimen is decreased on subsequent passes by rolling.

Comments on the Ductile-to-Brittle Transition Temperature

In tungsten, brittleness has been attributed to the presence of grain boundaries but not low angle boundaries.[21] Other work[10] has stated that sharply defined subboundaries have a higher ductile-to-brittle transition temperature than if diffuse subboundaries were present. Although somewhat indefinite, it does appear then that subboundaries should be avoided if possible. Room temperature ductility can be improved by warm working and thus increasing the dislocation

density. Purification if single crystals by zone refining also lowers the transition temperature; but, all of these gains are lost as soon as recrystallization occurs.[21] As a result, the lowest ductile-to-brittle transition temperature should be realized by retaining monocrystallinity in a high purity single crystal with a minimum amount of subgrains and a maximum amount of dislocation substructure. Since as-grown subboundaries are annihilated by small amounts of warm working[2] and some of the substructure retained when prior recovery anneals are used to raise the recrystallization temperature,[8] alternate rolling and annealing cycles under proper conditions should provide the most favorable form of tungsten for a low ductile-to-brittle transition temperature.

FUTURE WORK

1. Study the effects of prior recovery anneals on the recrystallization temperature of a 10 per cent rolled (100) [011] crystal which has been deformed at a temperature of 1000°C.

2. Observe as-grown subboundary annihilation, change in dislocation density and have Laue patterns to observe if the deformation is homogeneous and if it occurs by a simple glide.

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